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L7: Entry 2 of 2 File: USPT Jul 17, 1990

DOCUMENT-IDENTIFIER: US 4941976 A
TITLE: Dehydration of glycols

Brief Summary Text (11):
European Patent No. 0 096 3339 A2 to GFT as assignee of Bruschke discloses, as cross-linking agents, diacids (typified by maleic acid or fumaric acid); dihalogen compounds (typified by dichloroacetone or 1,3-dichloroisopropanol); aldehydes, including dialdehydes, typified by formaldehyde. These membranes are said to be particularly effective for dehydration of aqueous solutions of ethanol or isopropanol.

Brief Summary Text (38):
The preferred alphatic dialdehyde is glutaraldehyde. Aldehydes falling outside the scope of this invention typified by formaldehyde, glyoxal, or succinic semi aldehyde yield membranes which are characterized by unsatisfactory performance. Performance is judged by the ability of a membrane system to give a permeate containing less than 1 w % ethylene glycol (from a charge containing 85 w % ethylene glycol and 15 w % water) with a flux of at least 0.5 kilograms/meter.sup.2 hour (kmh) at a feed temperature of 80.degree. C. and with a permeate pressure of 5 mmHg and a condenser cooled by liquid nitrogen). Compositions falling outside the scope of this invention may be characterized by unsatisfactory selectivity or unsatisfactory productivity or both.

Brief Summary Text (39):
In situ crosslinking may be carried out by casting 5-10 w %, say 7 w % aqueous solution of polyvinyl alcohol which contains the aliphatic dialdehyde crosslinking agent. The mole ratio of crosslinking agent to polyvinyl alcohol may be 0.05-0.30, say 0.2.

Brief Summary Text (40):
Crosslinking is carried out, in the presence of acid catalyst, preferably inorganic acid. Sulfuric acid is preferred. Hydrochloric acid is much less preferred--because it yields membranes of poor selectivity, although the flux may be high.

Detailed Description Text (11):
From the above table, it is apparent that high concentration of crosslinking agent is needed in order to obtain highly selective membranes. At a mole ratio of 0.02 (Example VIII) the Selectivity is 20%; and as the mole ratio increases to 0.16 (Example XI) the selectivity desirably decreases, to 0.8. Commonly, it is desired to utilize a mole ratio of 0.1-0.2 say about 0.15 as this provides a good balance between selectivity and flux.

Detailed Description Text (18):
From the above table, it is apparent that putative crosslinking agents containing only one aldehyde group (Examples XV or XVII) or no aldehyde groups (Example XVI) are characterized by an undesirably low selectivity. Agents containing only two carbon atoms (glyoxal of Example XIV) are characterized by undesirably low selectivity. Example XVIII shows that use of hydrochloric acid as acid catalyst is much less satisfactory (than for Example XII) in that, it undesirably gives a much higher selectivity although the flux is significantly higher.

Detailed Description Paragraph Table (4):

TABLE

Membrane Performance				Crosslinking Agent	Acid Catalyst	Selectivity % EG	Flux kmh
XII	glutaraldehyde	sulfuric	0.28	0.48			
XIII	2-hydroxyhexane-dial	sulfuric	0.18	0.30			
XIV	glyoxal	sulfuric	2.61	0.16			
XV	formaldehyde	sulfuric	4.01	0.71			
XVI	succinic acid semialdehyde	sulfuric	3.04	0.76			
XVII	succinic acid	sulfuric	8.71	0.75			
XVIII	glutaraldehyde	hydrochloric	1.75	0.87			

Current US Cross Reference Classification (2):

210/500.42